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METHODS FOR DETERMINATION OF ACIDITY AND ALKALINITY IN HALOGENATED ORGANIC SOLVENTS AND THEIR ADMIXTURES

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Indian Standard

METHODS FOR DETERMINATION OF ACIDITY AND ALKALINITY IN HALOGENATED ORGANIC SOLVENTS AND THEIR ADMIXTURES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 12 June 1979, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 Different methods are employed for determining the acidity and alkalinity in halogenated organic solvents and their admixtures. This standard has been prepared keeping in view the methods which are generally followed.

0.3 In the formulation of this standard assistance has been derived from the following publications:

ASTM D 2989-1974 Part 30: Test method for acidity-alkalinity of halogenated organic solvents and their admixtures. American Society for Testing and Materials, USA.

ASTM D 2106-1964 (Reapproved 1971) Part 30: Test method for amine acid acceptance of halogenated organic solvents. American Society for Testing and Materials, USA.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods for the determination of acidity and alkalinity in halogenated organic solvents and their admixtures.

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*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. DETERMINATION OF ALKALINITY

3.1 Method A — *p*H Method

3.1.0 *Outline of the Method* — A sample of pure halogenated solvent or admixture is tested for *p*H by this method. If the *p*H of the sample is above 7·0, the alkalinity content is determined. The alkalinity of halogenated organic solvents and their admixtures is determined after extraction into a volume of water preadjusted to a *p*H of 3·9 by titrating with standard acid to a *p*H of 3·9. The extraction is performed either by stirring the immiscible liquids mechanically or by shaking the immiscible layers of liquids manually.

3.1.1 *Apparatus*

3.1.1.1 *p*H *meter* — glass electrode type.

3.1.1.2 *Stirrer* — electric or air-driven.

3.1.2 *Reagents*

3.1.2.1 *Water* — Adjust the *p*H of 1 litre (or any convenient quantity) of water to 3·9.

3.1.2.2 *Standard hydrochloric acid solution* — 0·02 N. Dilute 1·8 ml of concentrated hydrochloric acid to 1 litre with water and standardize it before use.

3.1.3 *Procedure*

3.1.3.1 *By using mechanical agitation* — Transfer 50 ml of the halogenated organic solvent to a 150-ml beaker and add 50 ml of water previously adjusted to *p*H of 3·9. Insert a stirrer, and stir as vigorously as possible for 10 seconds. Stop the stirrer and lift it out of the beaker. Allow the liquids to separate. Insert the *p*H meter electrodes into the separated aqueous upper layer, avoiding contact of the electrodes with the solvent layer, and titrate the water layer with hydrochloric acid to a *p*H of 3·9. When end point has been reached, remove the electrodes and reinsert the stirrer. Stir vigorously for 10 seconds again. Remove the stirrer, insert the electrodes, and again titrate to a *p*H of 3·9. Repeat the steps until additional stirring does not result in a change of *p*H. Record the final titration and calculate the alkalinity.

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*Specification for water for general laboratory use (*second revision*).

3.1.3.2 *By using hand agitation* — If no stirrer is available, the extraction and tiration may be carried out as follows:

Transfer 50 ml of the halogenated organic solvent to a 250-ml conical flask, add 50 ml of water preadjusted to *p*H of 3·9, and stopper the flask. Shake vigorously for 5 seconds. Pour the contents into a 150-ml beaker and allow the immiscible liquids to separate into layers. Insert the *p*H meter electrodes and titrate the water layer with hydrochloric acid to *p*H of 3·9. When the end point has been reached, remove the electrodes, return the liquids to the flask, and extract again for 5 seconds. Repeat the titration. Repeat the extraction and titration until no change in *p*H results from additional shaking. Record the final titration and calculate the alkalinity.

3.1.4 *Calculation* — Calculate the alkalinity as equivalent sodium hydroxide in mass percent as follows:

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{(V \times N \times 0{\cdot}04)}{M} \times 100$$

where

V = volume in ml of hydrochloric acid required for titration of the sample,

N = normality of hydrochloric acid, and

M = mass in g of sample used (volume in ml times its relative density).

NOTE — Where the solvent is trichloroethane, the equation becomes as follows:

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{(V \times N \times 0{\cdot}04)}{(50 \times 1{\cdot}463)} \times 100 = V \times N \times 0{\cdot}055$$

3.2 Method B — Indicator Method

3.2.0 *Outline of the Method* — The alkalinity of the halogenated organic solvent is determined, after extraction into a volume of water preadjusted to bromothymol blue end point, by titrating with standard acid to the bromothymol blue end point.

3.2.1 *Reagents*

3.2.1.1 *Water* — Add bromothymol blue indicator solution (a drop for each 10 ml of water) to 1 litre (or any convenient volume) of water and adjust to bromothymol blue colour change end point.

3.2.1.2 *Bromothymol blue indicator solution (0·1 percent)* — Dissolve 0·1 g of dibromothymol sulphonphthalein in 1·6 ml of 0·1 N sodium hydroxide solution and dilute to 100 ml with ethanol.

3.2.1.3 *Standard hydrochloric acid solution* — 0·02 N (*see* **3.1.2.2**).

3.2.2 *Procedure* — Transfer 50 ml of the halogenated organic solvent to a clean 250-ml conical flask, and add 50 ml of water adjusted to the bromothymol blue end point. Stopper the flask and shake for 5 seconds. Allow the immiscible liquids to separate, and titrate the aqueous upper layer with hydrochloric acid to the bromothymol blue end point. Stopper the flask, and again shake for 5 seconds. If the end point has changed, titrate to the bromothymol blue end point again. Repeat until no change in indicator results when additional shaking is done. Record the titration and calculate the alkalinity.

3.2.3 *Calculation* — *see* **3.1.4.**

4. DETERMINATION OF ACIDITY

4.0 Outline of the Method — A sample of the halogenated solvent or admixture is tested for *p*H. If the *p*H of the sample is below 7·0, the free acid content of halogenated organic solvent or admixture (with the exception of 1, 1, 1-trichloroethane) is extracted with neutral distilled water and determined by titration using either a *p*H meter or an indicator.

4.1 Reagents

4.1.1 *Neutral Distilled Water* — Boil 1 litre of distilled water for 5 minutes in a borosilicate glass or stainless steel container, then cover and cool to room temperature. Titrate a 50 ml aliquot to *p*H of 7·0 to 7·3 with either 0·01 N sodium hydroxide solution or 0·01 N hydrochloric acid, depending upon whether the initial *p*H is above or below 7. From this titre, calculate the volume of 0·01 N sodium hydroxide solution or 0·01 N hydrochloric acid required to adjust the *p*H of the remaining 950 ml of boiled distilled water to *p*H of 7·0 to 7·3. Add the calculated volume of 0·01 N sodium hydroxide solution or 0·01 N hydrochloric acid together with the titrated 50 ml aliquot of neutralized water to the 950 ml stock and mix thoroughly. Determine the *p*H of a second aliquot. If further adjustment is necessary to obtain a *p*H of 7·0 to 7·3, repeat the above procedure.

4.1.2 *Bromothymol Blue Indicator Solution* — 0·1 percent (*m/v*) (*see* **3.2.1.2**).

4.1.3 *Sodium Hydroxide Ethanolic Solution* — 0·01 N. Dissolve 4 g of sodium hydroxide in anhydrous ethanol and dilute to 1 000 ml with ethanol. Standardize by any accepted procedure to determine the exact normality. Prepare 0·01 N ethanolic sodium hydroxide solution by diluting 10·0 ml of the standardized 0·1 N sodium hydroxide solution to 100 ml with anhydrous ethanol in a calibrated volumetric flask.

4.2 Procedure

4.2.1 *By pH Method*

4.2.1.1 Take 50 ml of halogenated organic solvent or admixture into 250-ml separating funnel containing 50 ml of freshly boiled neutral distilled water adjusted to the bromothymol blue end point. Shake the mixture for 2 minutes, then let stand and allow the layers to separate. Take a 25 ml aliquot of the water layer into a 100-ml beaker. Determine the acidity of the water extract by procedure given in **4.2.1.2**.

NOTE — The bromothymol blue end point may be interpreted as an aqua (blue-green) to light blue colour.

4.2.1.2 Titrate the sample while stirring with 0·01 N sodium hydroxide solution to a *p*H of 7·0 to 7·3. Record the titration and calculate the acidity as given in **4.2.2.2**.

4.2.2 *By Indicator Method*

4.2.2.1 Take 50 ml halogenated organic solvent or admixture into a 250-ml separating funnel containing 50 ml of freshly boiled neutral distilled water adjusted to the bromothymol blue end point. Shake the mixture for 2 minutes, then let stand and allow the layers to separate. Take a 25 ml aliquot of the water layer into a 100-ml beaker. Add 1 ml of bromothymol blue indicator solution and titrate with 0·01 N sodium hydroxide solution to the bromothymol blue end point. Record the titration and calculate the acidity.

4.2.2.2 *Calculation* — Calculate the acidity of halogenated organic solvent or admixtures as equivalent hydrochloric acid in mass percent as follows:

$$\text{Acidity (as HCl), percent by mass} = \frac{(V \times N \times 0{\cdot}036\,5 \times 2)}{M} \times 100$$

where

V = volume in ml of sodium hydroxide solution required for titration of the sample in **4.2.1.2** or **4.2.2.1**,

N = normality of the sodium hydroxide solution, and

M = mass in g of sample used (volume in millilitres times its relative density).

4.2.3 *Procedure for 1:1:1 Trichloroethane* — Pipette 50 ml of 1,1,1-trichloroethane into a clean, dry, 100-ml conical flask. Add 1 ml of bromothymol blue indicator solution and titrate with the 0·01 N ethanolic sodium hydroxide solution to the bromothymol blue end point.

4.2.3.1 *Calculation* — For 1,1,1-trichloroethane or admixtures, calculate as follows:

$$\text{Acidity (as HCl), percent by mass} = \frac{(V \times N \times 0{\cdot}0365)}{M} \times 100$$

where

V = volume in ml of sodium hydroxide solution required for tiration of the sample,

N = normality of the sodium hydroxide solution, and

M = mass in g of sample used (volume in millilitres times its relative density).